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Dry Coating In A High Shear Mixer: Comparison Of Experimental Results With DEM Analysis Of Particle Motions

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Abstract. Experimental dry coating of guest particles on the surface of host particles is performed by mechanical forces in a high shear mixer called "Cyclomix". The studied system (a mixture of particles of sugar, "Suglet™" as host particles and magnesium stearate as guest particles) was chosen as a model one to achieve better understandings of the phenomena during mixing. To simulate the flow of host/guest particles in the mixer, the Discrete Element Method (DEM) was applied. Experimental results such as flowability and wettability can be explained by particles flows evolutions with different rotational speed or duration treatment inside the Cyclomix.

Keywords: Dry coating, Mixer, DEM, particle motion
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INTRODUCTION

Dry particle coating is a process allowing designated modifications of host particles properties. A coating of fine guest particles onto the surface of host particles is achieved using mechanical forces such as shear and impact forces, generally occurring during mixing. This process has received a widespread attention by many industrial fields because it consists in an environmental friendly and low cost operation avoiding organic solvents. The optimization of a dry coating process requires normally the experimental efforts and empirical techniques [1-3]. Several theoretical approaches have been reported [4,5].

Meanwhile, the Discrete Element Method (DEM), proposed by Cundall and Strack [6], let us trace precisely the motion of each particle. The DEM is one of the most popular numerical methods for simulating and analyzing the solid particle behavior and has been successfully applied in many fields such as soil and rock mechanics [7], agricultural product handling [8], storage and flow of granular materials [9] and so on. DEM has already been applied to several studies involving other dry coating apparatus [10,11]. CFD modeling of particles motion inside the Cyclomix has been applied for granulation experiments [12, 13].

This work contributes to apply DEM to the simulation of the particle motion in a high shear mixer, and its analysis regarding the evolutions of physical properties obtained by experimental work.

EXPERIMENTAL

Sample Powders

The sample powders chosen are sugar particles (Suglets™) and magnesium stearate (MgSt) for host and invited particles respectively. Suglets, products of Colorcon INC., are spherical cores mainly composed of sucrose and maize starch with a highly hydrophilic character. MgSt supplied by Chimiray chosen as invited particles is a fine, white, cohesive and hydrophobic powder. The size distribution of Suglets seems to have a one shape population with median diameter (D50) of about 250 µm, and indicates that this powder has a very homogeneous size, in comparison with that of MgSt having a wide population ranging from 0.1 µm to 50 µm. D50 of MgSt is about 5 µm.

Coating Device

The coating device used in the experiment is a high shear mixer (Cyclomix, Hosokawa micron B.V. Japan), which is generally used for granulation. This device has been successfully used for dry coating [14]. The schematic diagram of the experimental equipment is shown in Fig. 1. This device presents basically a stationary conical chamber and a vertical rotor that can rotate clockwise direction from 200 rpm to 3000 rpm, with four pairs of flat-bladed impellers from bottom to top. Free volume of the chamber is 1000 cm^3.

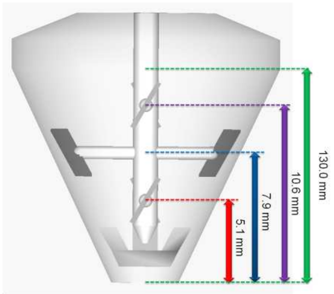

FIGURE 1 : Schematic diagram of the high shear mixer.

RESULT AND DISCUSSION

Numerical analysis

The particle motion in the Cyclomix has been simulated by the discrete element method (DEM).

The particles with the properties of the host particles given in Table 1 were placed in the vessel of the Cyclomix with an hexagonal-close packed structure.

TABLE 1. Simulation parameters.

Young's modulus	E [GPa]	4.5
Poisson's ratio	[-]	0.30
Density	[kg/m3]	1600
Coefficient of restitutuion	[-]	0.16
Time step	Δt [µs]	2.0
Total simulation time	T [s]	1.5

Gravity effects have been introduced as an external condition imposed to the assembly yet submitted to the forces developed by the rotating impellers. The current computer power is not enough to simulate the actual number of particles. Then a smaller number of particles (about 40 000), but with larger size (2.0 mm) has been chosen.

Velocity and motion analysis

The particle motion, as well as the velocity of each particle has been analyzed from the snapshots of the simulations with different operating conditions. The color shows the normalized dimensionless velocity (red represents 1 and blue represents 0).

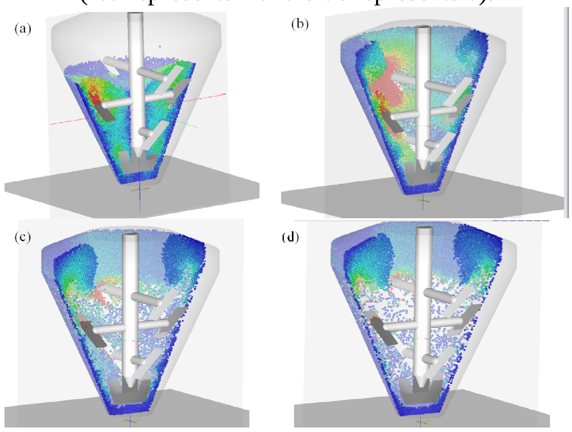

FIGURE 2. Normalized velocity of particle in color at each rotational speed: (a) 100 rpm, (b) 500 rpm, (c) 1000 rpm and (d) 3000 rpm, particle size 2 mm, filling ratio J = 60 % in cross section.

Fig.2 shows the normalized velocity of a particle in color at each rotational speed: (a) 100 rpm, (b) 500 rpm, (c) 1000 rpm and (d) 3000 rpm in cross section views. In the cross section, the particle motion is revealed clearer, at low rotational speed (100 rpm), the particle bed stays below the top impeller, and particles around paddle have relatively high normalized velocity. At 500 rpm, the powder bed reaches the top of the chamber, and at 1000 rpm, most of the particles go to the upper side. Finally at 3000 rpm, the particles stay either on the upper side or bottom, and there are only few particles around the middle part of the chamber.

Simulation of host and guest particles without adhesion

The simulations have been performed in order to know the effect of rotational speed on the mixing of host and guest particles. The rotational speed varies from 250 to 1500 rpm. The filling ratio of samples is fixed at 40 % and 20 % for host and guest particles, respectively. Simulation time is also fixed at 3.0 s. Particle size is also fixed at 3 mm and 1 mm for host and guest particles respectively. The physical parameter used for the host and guest particles are summarized in Table 2. Initially the invited particles are located over the powder bed of host particles.

TABLE 2. Physical characteristics of host and guest particles

material		host	guest
Young's modulus	E [GPa]	4.5	2.2
Poisson's ratio	[-]	0.30	0.30
Density	[kg/m3]	1600	1120
Coefficient of restitution	[-]	0.3	0.0
Number of particles	[-]	14 148	190 986
Total simulation time	T [s]	3.0	

To observe the inside of the mixing chamber, the cross sections of the mixing chamber at each rotational speed are shown in Fig. 3 (Guest in Blue and host in Red). At 250 rpm the guest particles are located around the surface of the powder bed and rotating axis (Fig. 3 (a)).

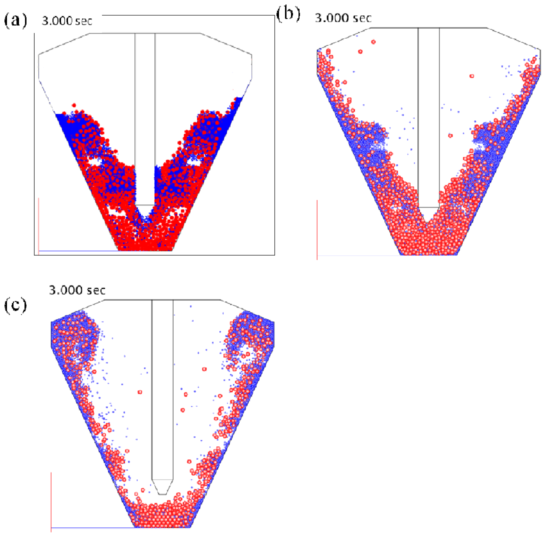

FIGURE 3 : Cross section of the mixing chamber at each rotating speed: (a) 250 rpm, (b) 500 rpm, (c) 1500 rpm, T= 3s (host in red and guest in blue).

At higher rotational speeds, 500 rpm, the guest particles are located along the side wall (Fig. 3 (b)). It is suggested that the dry coating occurs in this region. At 1500 rpm the guest particles are evenly located around the side wall and especially there are a lot of guest particles seen at the upper part of the mixer (Fig. 3 (c)). Since the guest particles are smaller and lighter than the host particles, they are likely to go upper side.

Discussion

The sessile water drop test has been carried out to analyze the wettability of the products. Fig. 4 shows the contact angle measured 30 s after the water drop is placed on the powder surface for different processing times t, different speeds of rotation ω and at a filling ratio of J = 60 % .

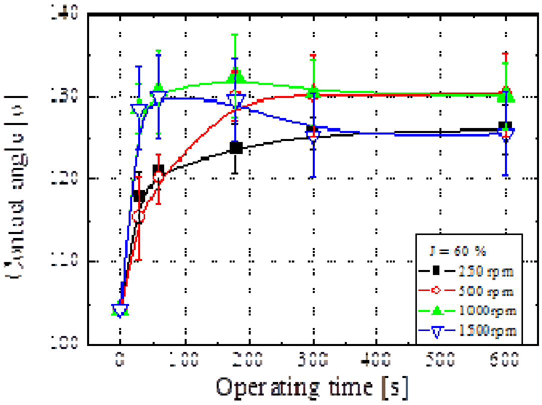

FIGURE 4 : Dependence of the contact angle on the operating time t, and on the different rotational speeds ω for a filling ratio J = 60%.

In Fig. 4, at 250 rpm, the contact angle increases with an increase of processing times. From the assumption above, the surface fraction of the coated material will also increase. In other words, the coating process proceeds as a function of the processing time; at 500 rpm, it increases more rapidly than at 250 rpm. At 1000 rpm, $\theta(t)$ increases with time and is asymptote to the contact angle θ_l of guest particles. At 1500 rpm, it increases until 180 s and then starts to decrease gradually. One explanation of this drop of contact angle is that new surfaces of suglets not covered by MgSt are created. It could be said that once fine fragments of the host particles are generated by the attrition or erosion during the coating operation, after that, those fine fragment of host particles stick to the surface of the host particles again. The contact angle and surface fraction of coated particles could decrease at high speed coating operation by those phenomena.

Regarding the evolutions of particles motions found by DEM, we will try to explain the evolution of physical properties of composite powders formed by mixing inside the cyclomix. All experimental results are explained [15,16]. We have divided the coating process inside the Cyclomix into several steps strongly depending on the rotational speed.

The first step of discrete coating is especially occurring at low rotational speeds. The evolution of wettability is very slow and the maximum contact angle showed in fig 4 for 250 rpm do not reach the value of 132°, so we can tell that the coating is not complete at the surface of suglets. If we look at particle motions in fig 2 and 3 we can see that the particles are not individualized and packed at the lower part of the cyclomix. So, it takes a long time for small particle to stick on the surface of big particles.

For these small rotational speeds we cannot achieve complete coating due to insufficient mixing of both powders clearly shown in Fig 3a.

For rotational speed of 500 rpm and 1000 rpm, the complete coating is achieved and the properties do not vary at a steady state step. Wettability of powders reached the same value for a 10 minutes mixing time. If we look at the particle motions, the powder bed reaches the top of the Cyclomix Fig 2b) and Fig 2c) and the fig 2b) shows that that the small particle are on the wall of the Cyclomix. So the coating can occur at these rotational speeds and is be located along the walls of the Cyclomix.

For higher rotational speed (faster than 1000 rpm), a particle breakage can occur; this is shown with the drop of contact angle from 132° to 125°. The particle motions were quite the same for 1000 rpm and 3000 rpm and the coating occurs rapidly (in less than one minute). After that, this higher energy given to the system could generate brakeage and / or attrition of suglets [15,16]. For this host/invited particles system these rotational speed are not good for dry coating.

CONCLUSIONS

In this paper, the particle motion of particles in a high shear mixer has been simulated by DEM. The validation of the simulation work was discussed in comparison with experimental data (such as wettability). The velocity of the particles simulated by the DEM can explain the behavior of the coating and helped us to better understand several phenomena. The coating at low rotational speed was not enough efficient because the small particles were not really mixed to the larger particles and they still remain stuck together in large agglomerates. For intermediate rotational speed, the coating occurred and seemed to be located along the Cyclomix walls. For higher rotational speeds, the energy given to the particles was high enough to generate breakage.

These results confirm the applicability of the proposed method for simulating the particles motions in the dry coating. This methodology should contribute to the evaluation of the dry particle coating efficiency from mechanical studies (i;e. calculate contacts forces of particles / particles / impellers/wall) of characterization of particles contacts in order to extend predictions to other coating systems.

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